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Australian Nuclear Science & Technology Organisation

Evolution of composition and grain correlations upon phase transitions and micro-structural rearrangement processes followed in-situ by high-energy X-ray diffraction

Klaus-Dieter Liss

neutrons ... science for Australia

materials AUSTRALIA

International Conference and Exhibition

Materials and Austceram

4th - 6th July 2007, Sydney, Australia

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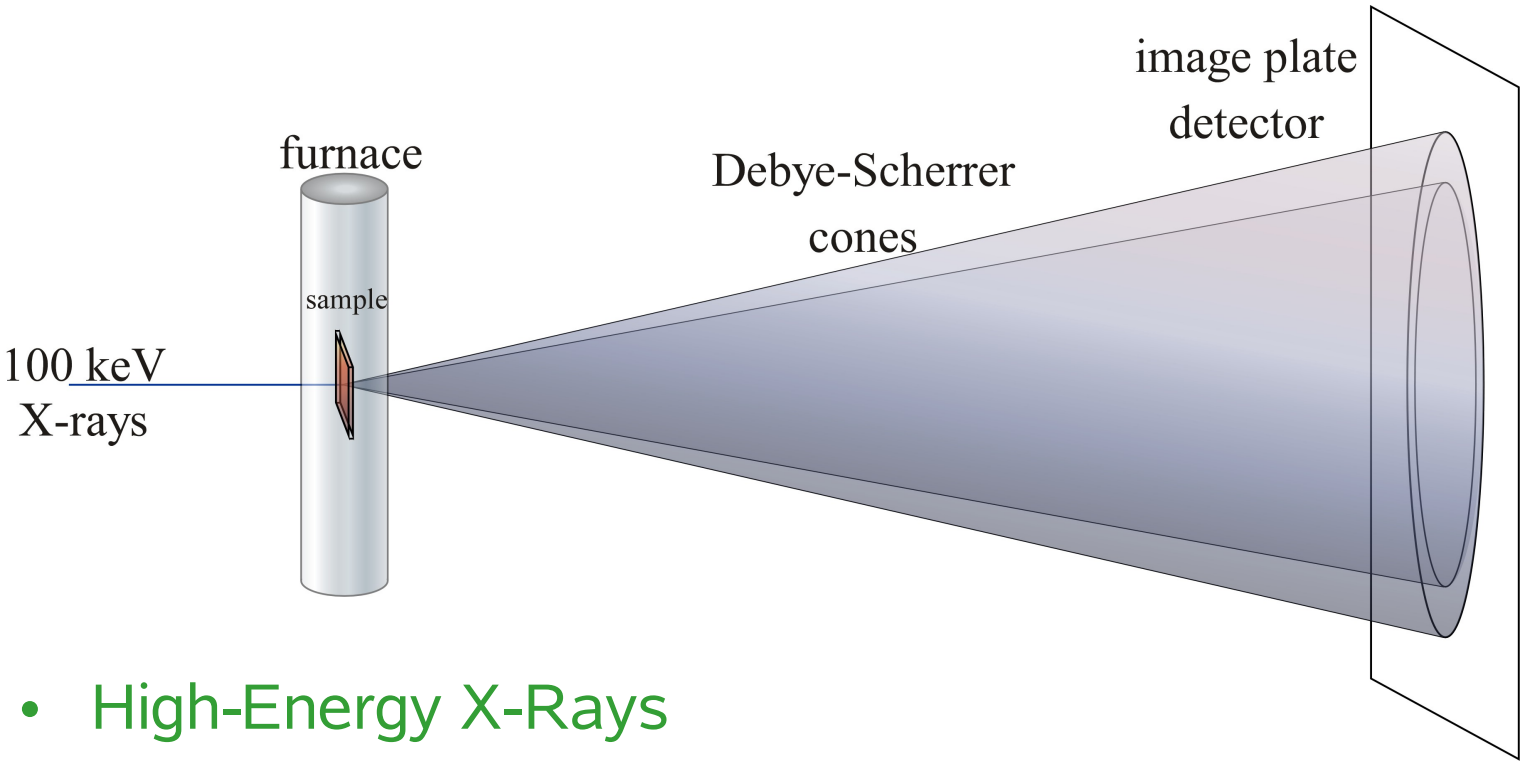
Overview

- In-situ techniques
  - High Energy X-ray diffraction - 2D
  - Laser Scanning Confocal Microscopy
- Ti-Al system
- In-situ experiments
  - massively transformed γ-sample
  - α_2-rich sample
- Interpretation
- Results

- Collaborators

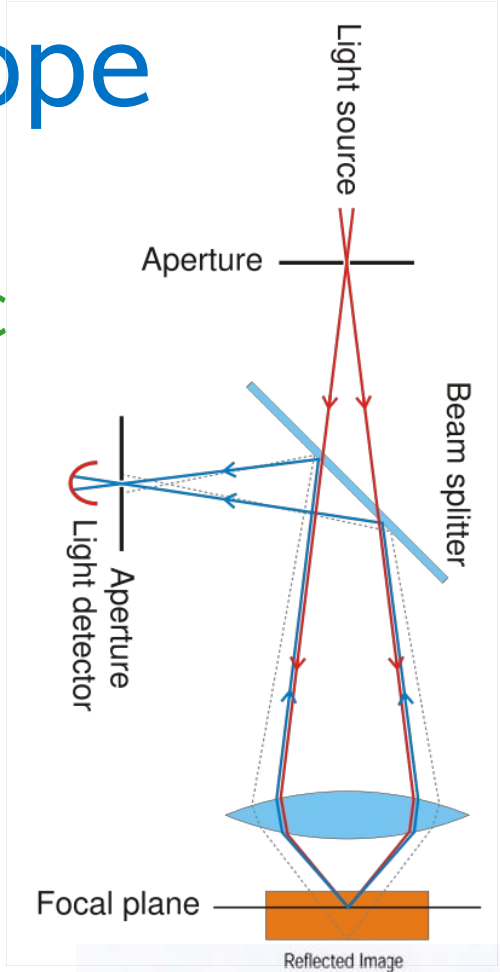
Registration of Debye-Scherrer Cones

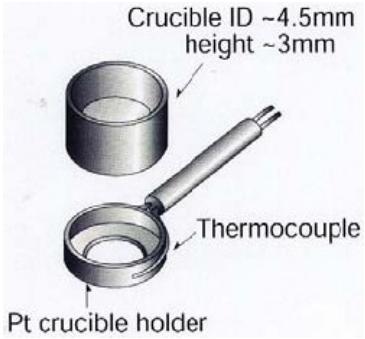


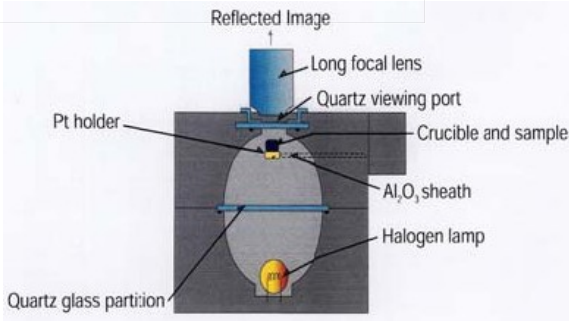
- High-Energy X-Rays
- Penetration ➡ bulk
- Very simple setup
- High G-vectors
- Operates in air / argon
- Sample environment
- 2D-detector
- Fast data acquisition

Laser Scanning Confocal Microscope

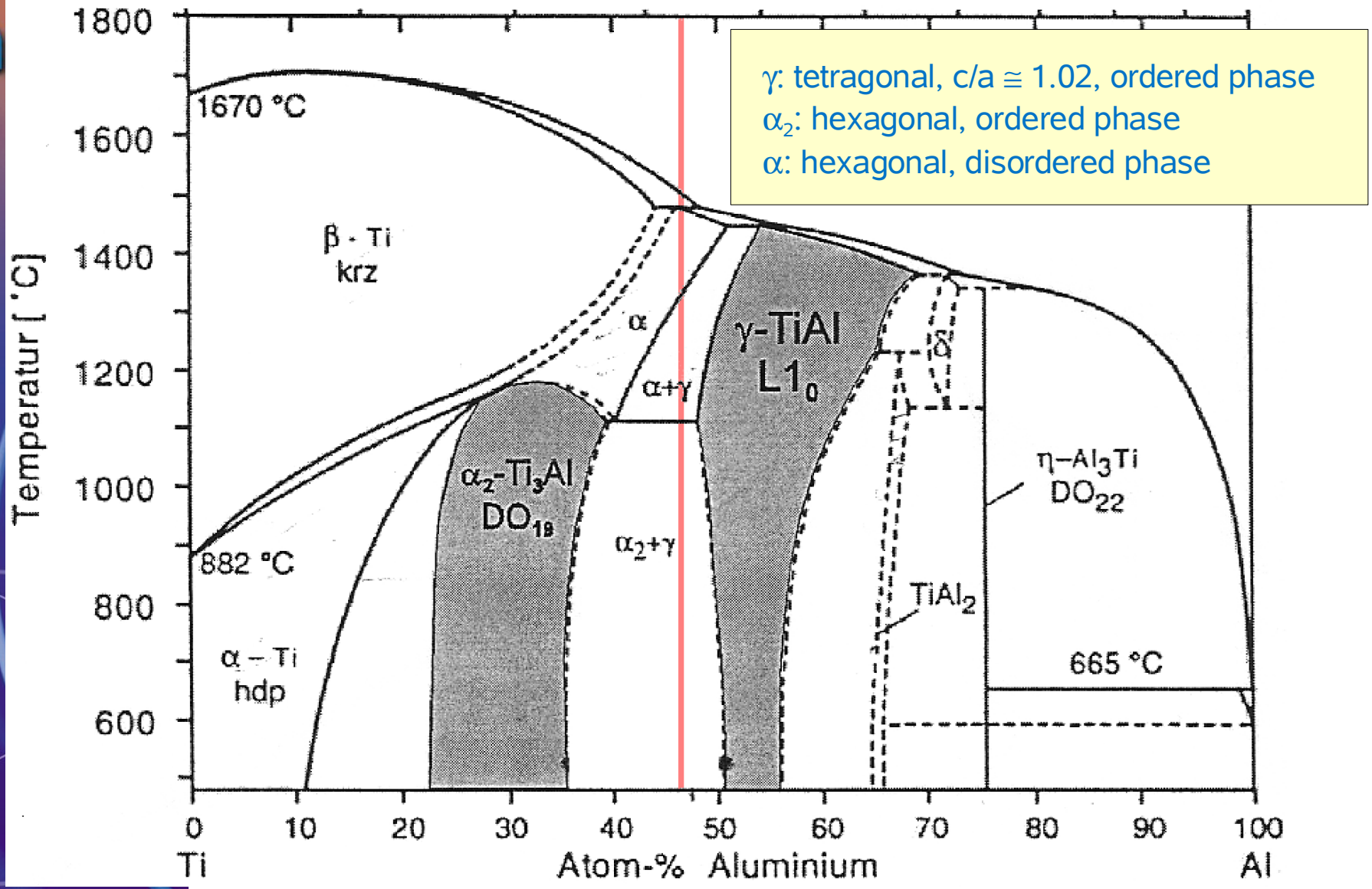
- sees steps in sample surface
- in-situ heating device 2000 °C
- discrimination from thermal radiation
- real time image
- CCD camera 30 frames / sec



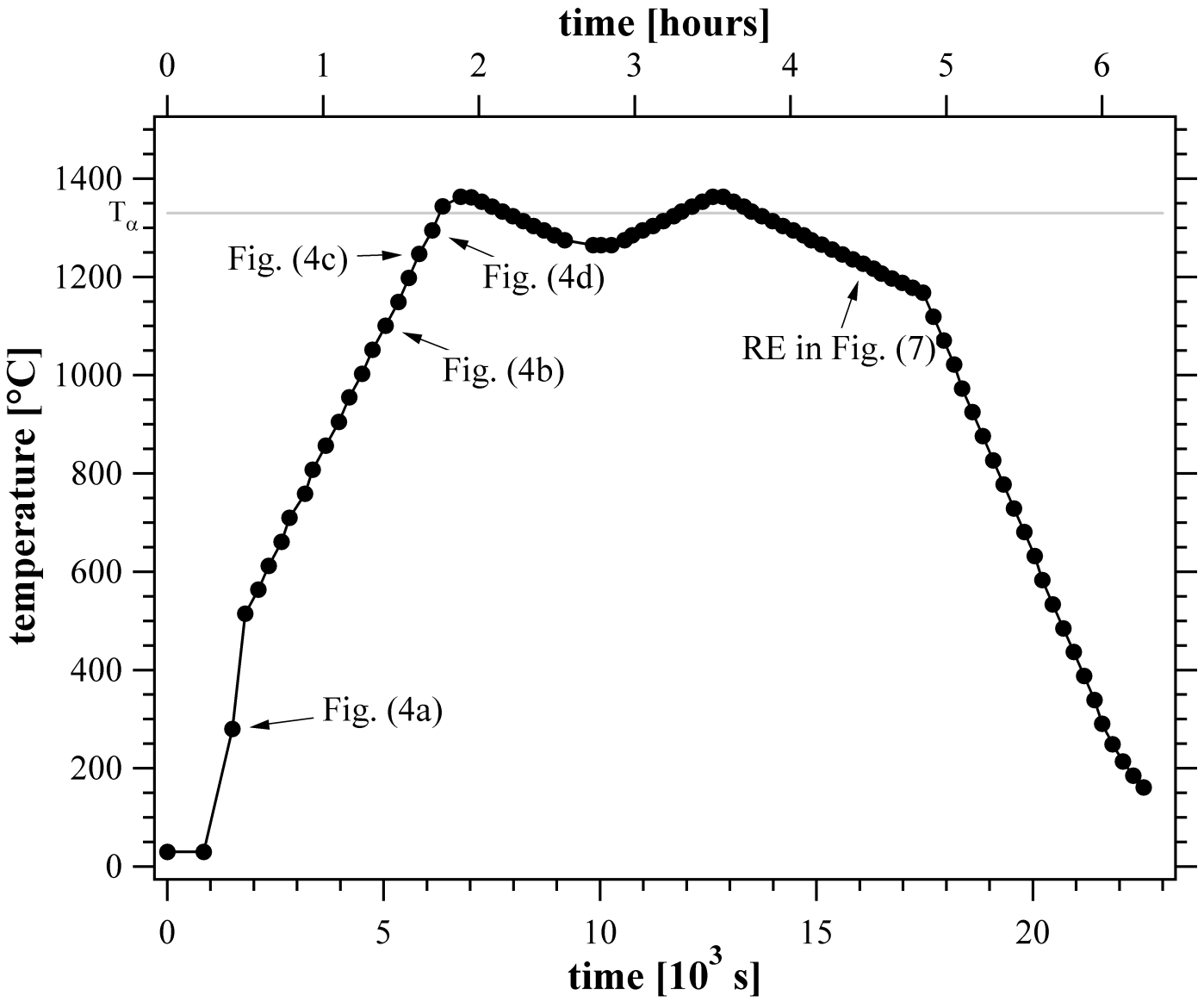




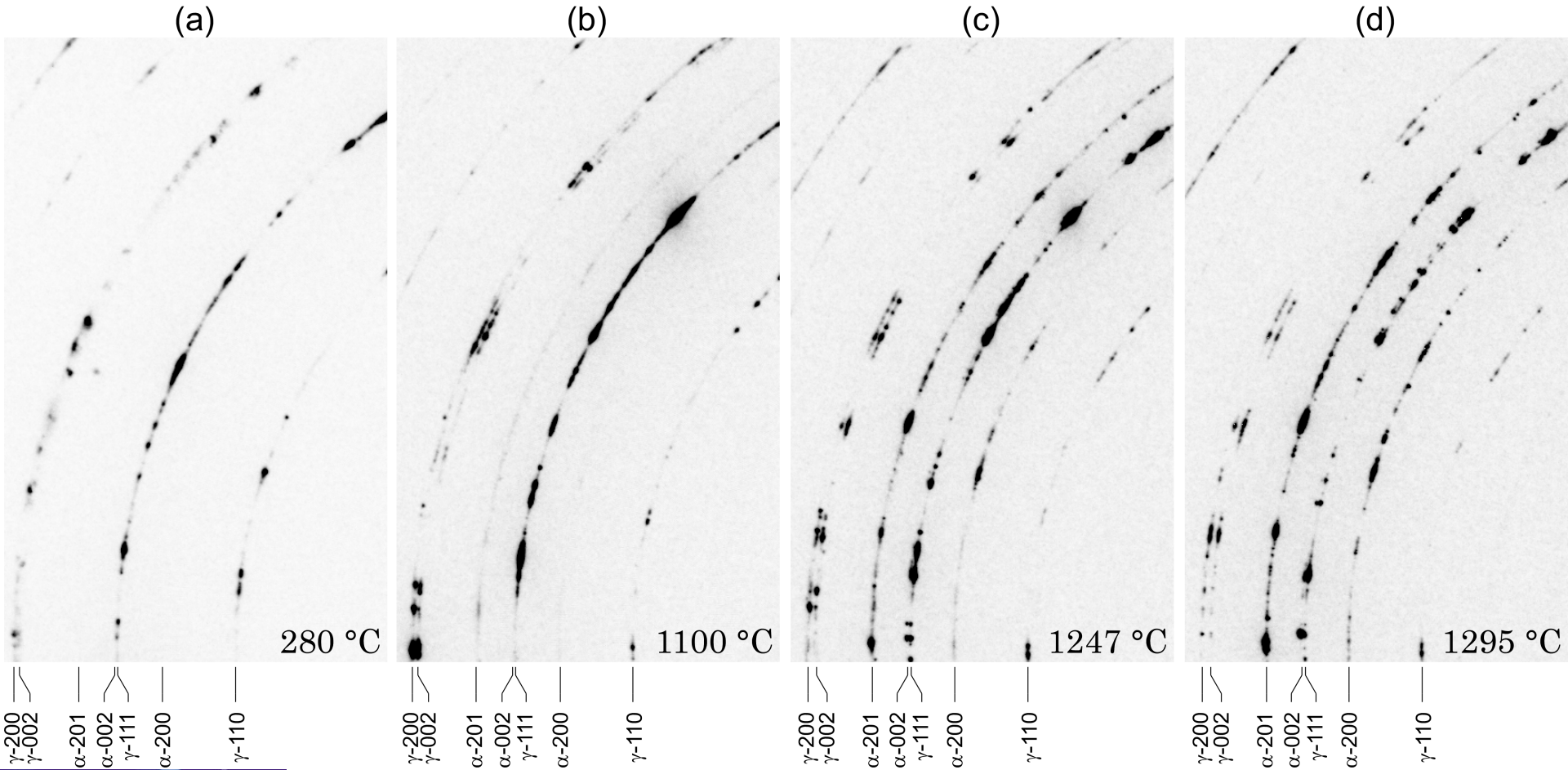
Phase diagram of Ti-Al



Heating Cycle

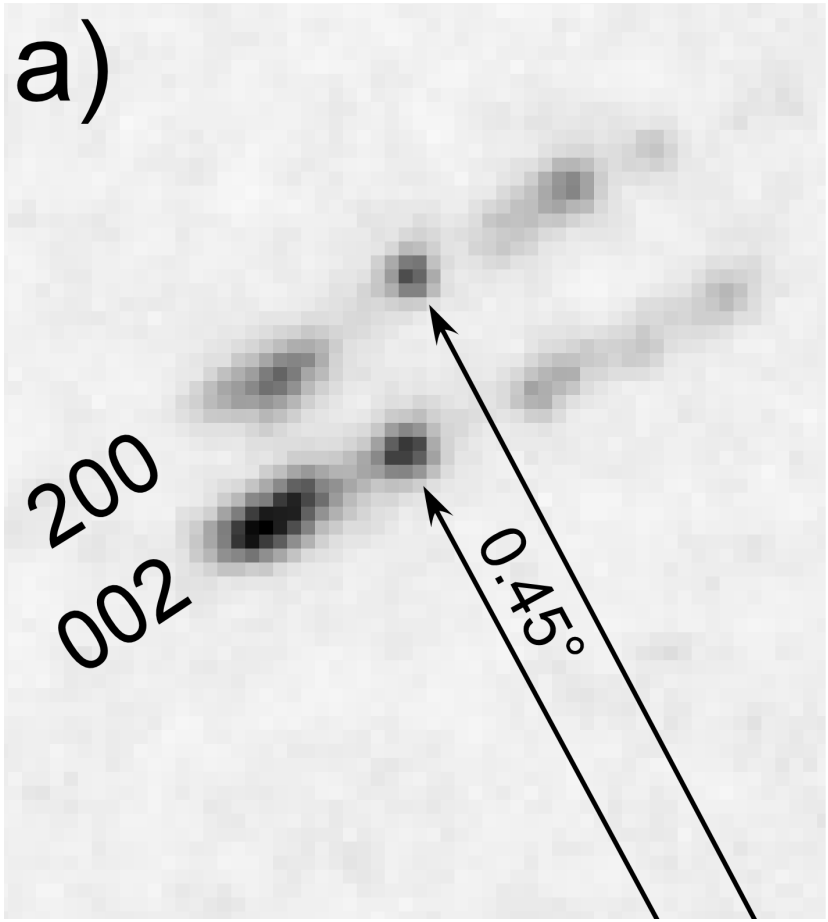


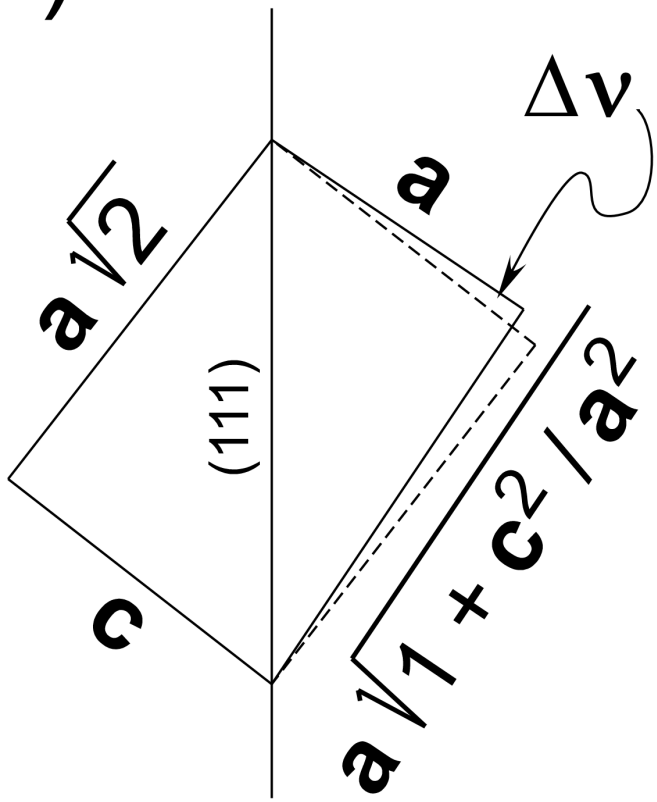
Ramp-Up heating



Massively Transformed γ-TiAl

Correlated Reflections

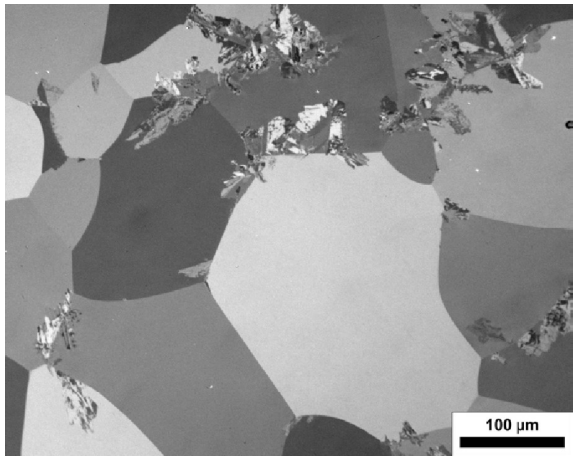




domain boundary

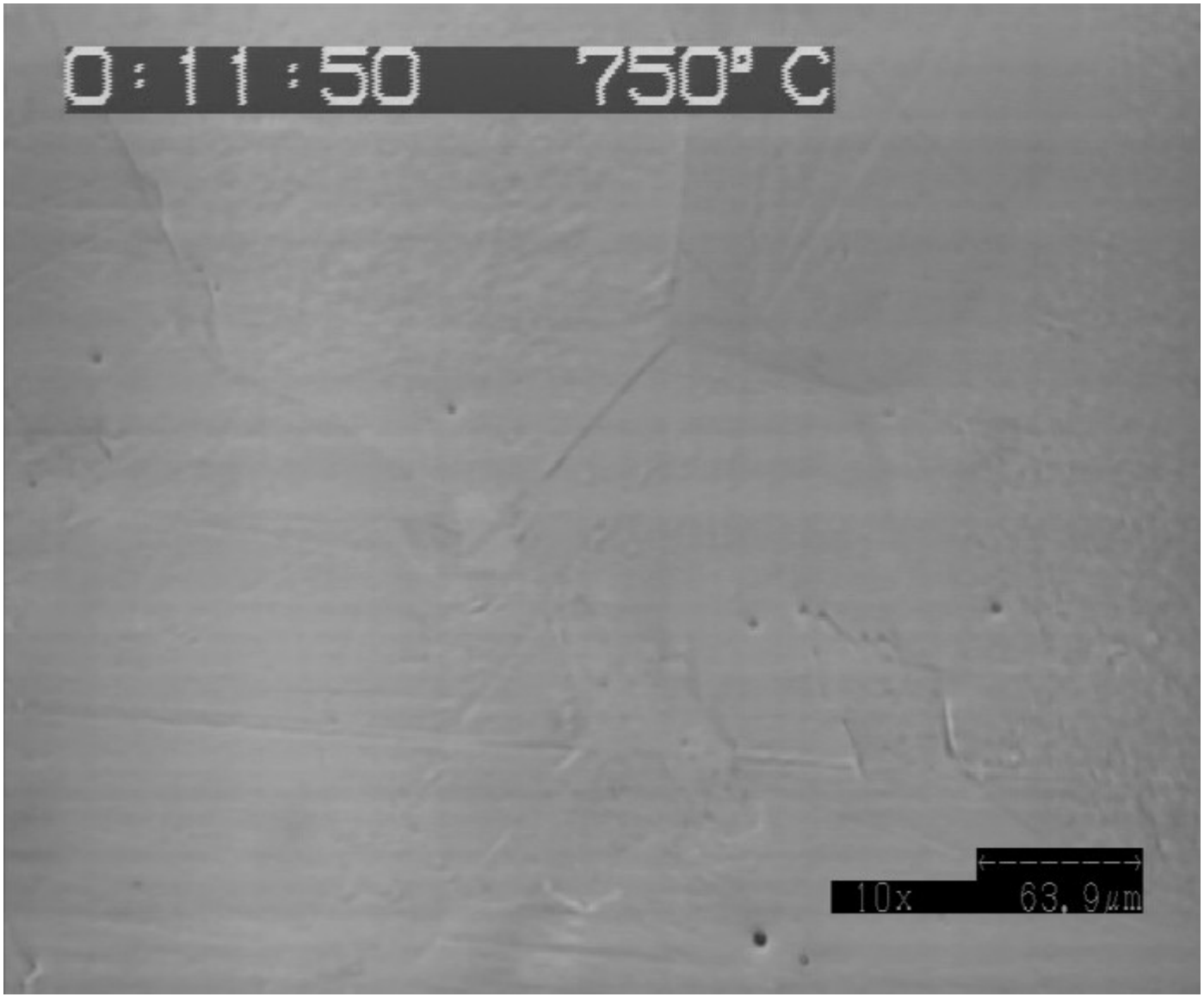
Time resolved in-situ experiments

- Start with α_2-rich (AR) sample
- Transform into $\gamma(+\alpha_2)$ upon heating
- Transform back into $\alpha(+\gamma)$ upon further heating
- Transform into pure α above 1300°C



Preparation:
- 5 min @ 1320 OQ
- 90% α/α_2
- 10% MT γ

In-situ cycle: LSCM



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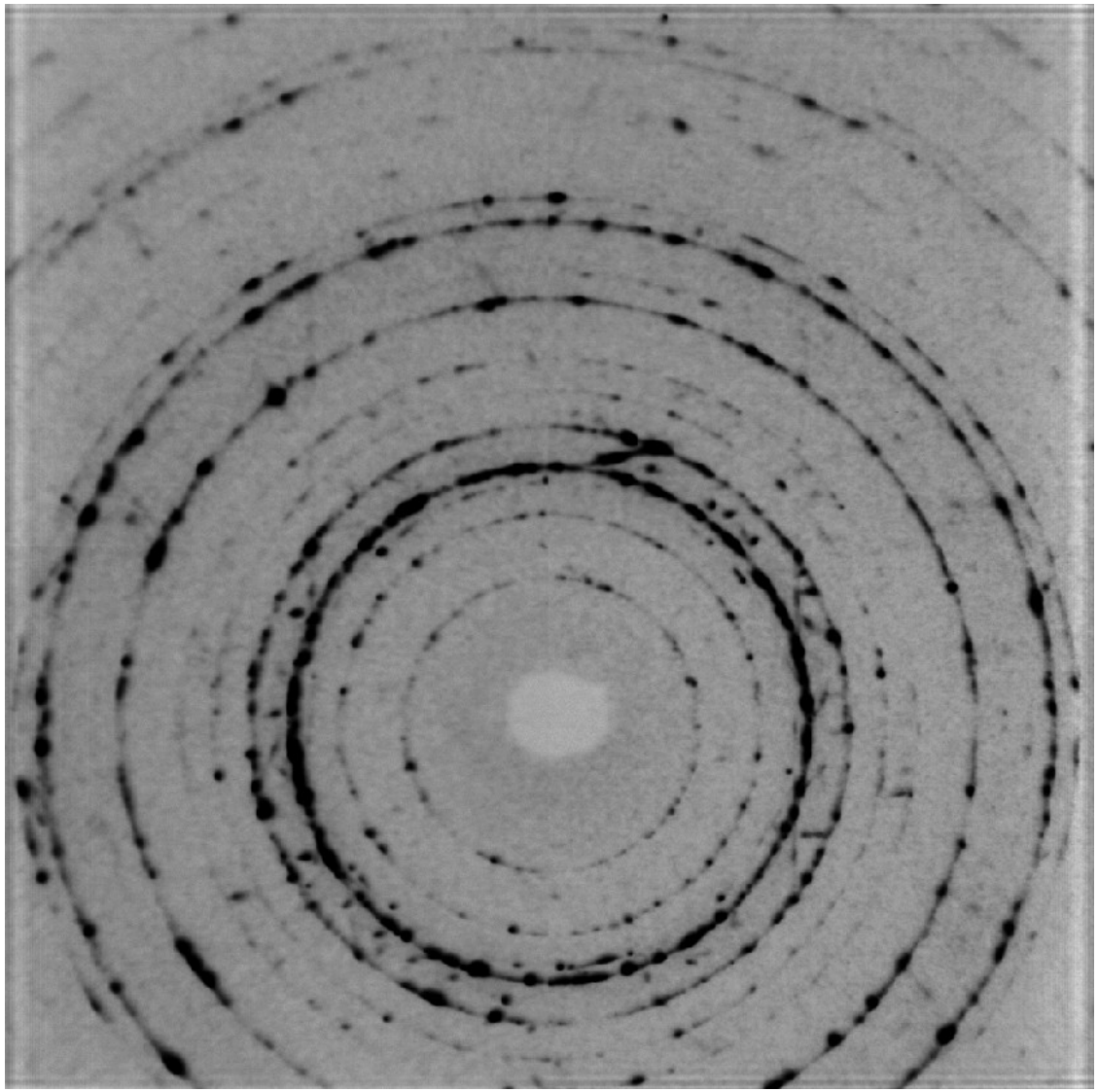
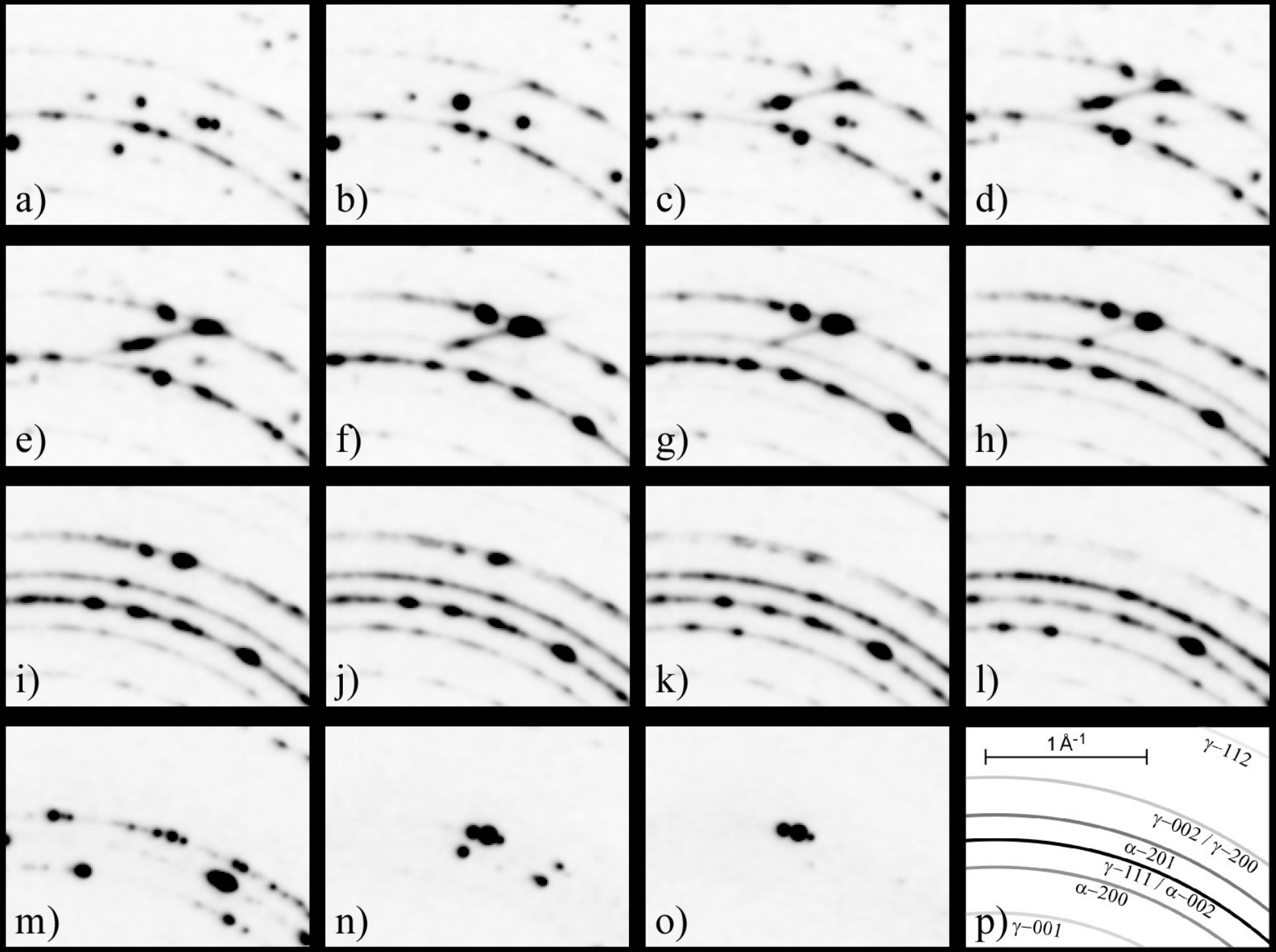
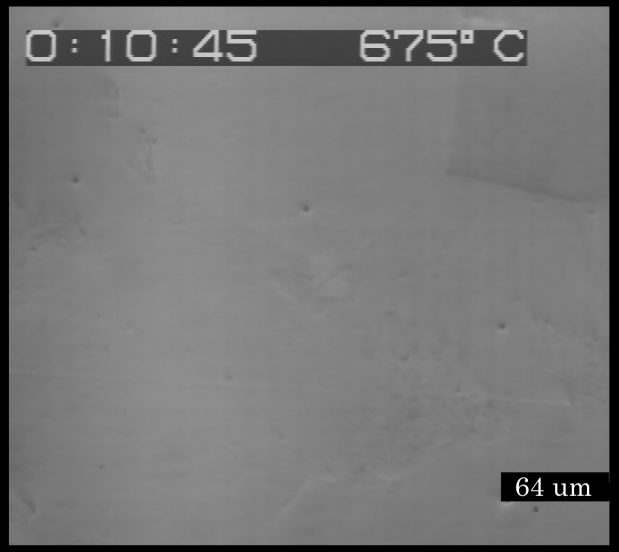
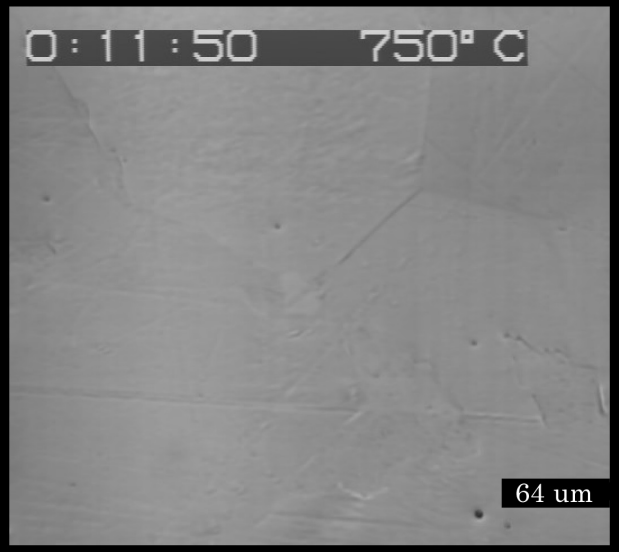
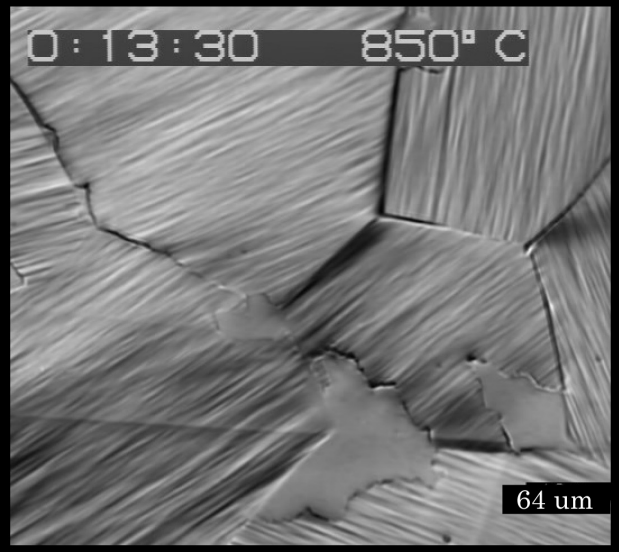
In-situ cycle: diffraction

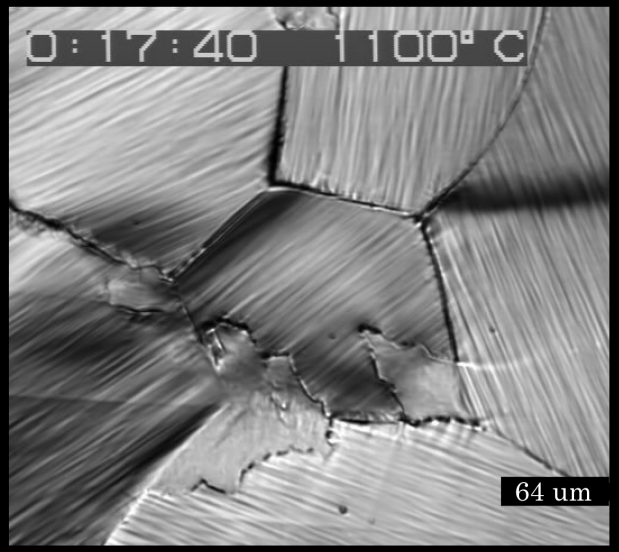


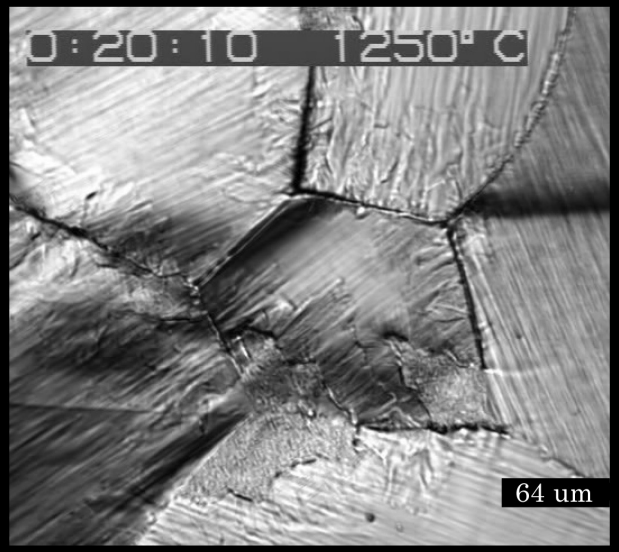

a)
b)
c)
d)
e)
f)
g)
h)
i)
j)
k)
l)
m)
n)
o)
p)
1 Å-1
γ–112
γ–002 / γ–200
α–201
γ–111 / α–002
α–200
γ–001

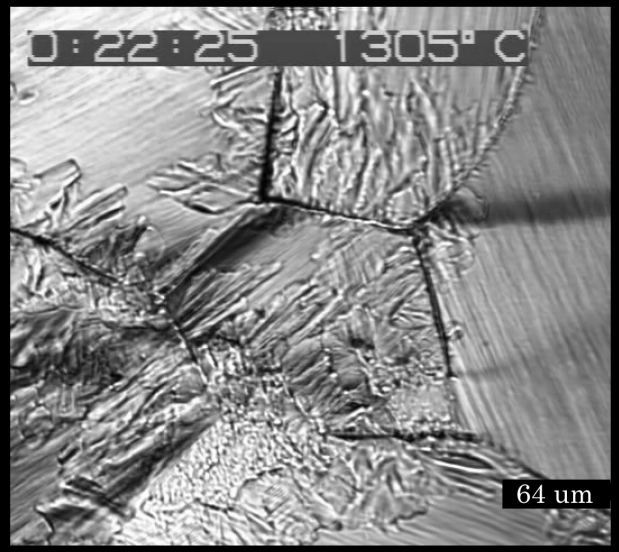

0:10:45 675° C
64 um

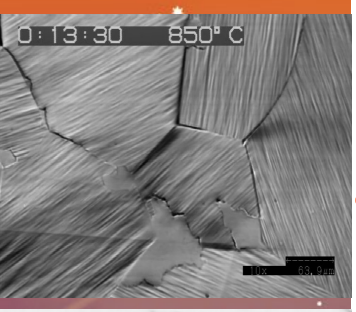

0:11:50 750° C
64 um

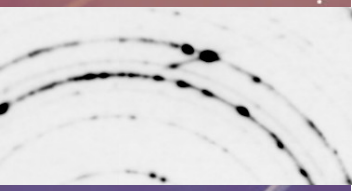

0:13:30 850° C
64 um

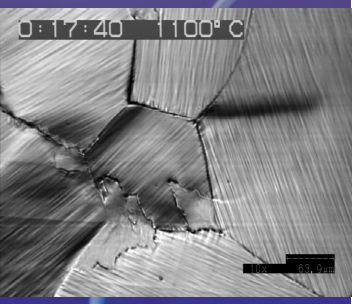

0:17:40 1100° C
64 um

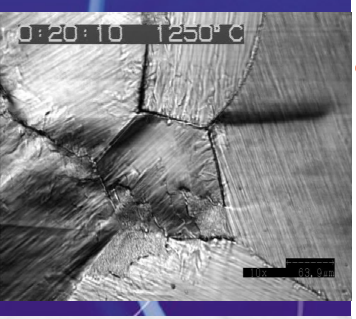

0:20:10 1250° C
64 um

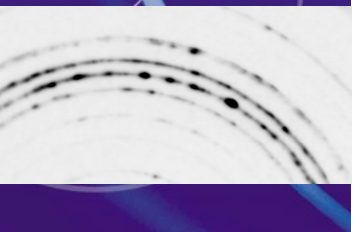

0:22:25 1305° C
64 um











Structural Transformations

- quenched α/α_2 ➡ $\alpha_2+\gamma$ (600 – 1100°C)
  - $Ti_{54}Al_{46}$ ➡ Ti_3Al + TiAl
  - orientation relation: 1 possibility α-{002} || γ-{111}
  - appears in the bulk
  - ultra fine, local lamellae
  - needs structural rearrangements
  - needs segregation
  - occurs through gradient in orientation and lattice spacing
  - well ordered, coherent transition
- $\alpha_2+\gamma$ ➡ $\alpha+\gamma$ (1100 – 1300°C)
  - Ti_3Al + TiAl ➡ Ti_3Al + TiAl
  - orientation relation: 4 possibilities γ-{111} || α-{002}
  - nucleates on grain boundaries
  - irregular growth, starts well correlated, grain refinement
  - blocks grain growth until γ disappears above 1300°C

Results

- novel in-situ studies in real time
  - diffraction + microscopy
  - 'bad' 2D powder diffraction patterns reveal crystallographic correlations

- phase transitions are well ordered and directional
- homogeneously over bulk (α_2 ➡ γ)
- nucleation on grain boundaries (γ ➡ α)
- diffuse streak: lattice gradient + phonons?

Collaborators

TU Hamburg-Harburg (TUHH), Germany

Dr. Arno Bartels, project group leader for TiAl at TUHH
Slawomir Bystrzanowski, sample preparation, metallurgy
Andreas Stark, metallurgy

Montanuniversität Leoben, Austria

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Prof. Dr. Rian Dippenaar, institute head, LSCM

GKSS research center, Geesthacht, Germany

Dr. Rainer Gerling, group leader TiAl, production and science
Dr. Frank-Peter Schimansky, TiAl production and metallurgy

European Synchrotron Radiation Facility (ESRF), Grenoble, France

Dr. Thomas Buslaps, beamline operation manager, diffraction setup

Bragg Institute:

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